METHOD 7474

MERCURY IN SEDIMENT AND TISSUE SAMPLES BY ATOMIC FLUORESCENCE SPECTROMETRY

1.0 SCOPE AND APPLICATION

1.1 This procedure measures total mercury in sediment and tissue samples.

1.2 The range of this method is from approximately 1 part per billion to the part per million range. Analysis of the entire range cannot be accomplished at once, but rather different portions of this range can be analyzed depending upon the instrument gain settings.

1.3 This method should only be used by analysts experienced with the analysis of trace elements at very low concentrations when analyzing samples in the ppb range.

2.0 SUMMARY OF THE METHOD

2.1 A representative portion of sample is digested in a microwave unit (a variation of Method 3052) using nitric and hydrochloric acids in a closed fluorocarbon container. The sample is digested under pressure to aid in the dissolution of organic compounds containing mercury.

2.2 An aliquot of the digested sample is diluted and subjected to cold digestion with an acid/bromate/bromide mixture.

2.3 Stannous chloride is added to the digested sample as a reducing agent to produce Hg^0. The reduced mercury is separated from the sample/reagent mixture as a vapor that is carried to the fluorescence detector by a stream of high purity argon.

3.0 DEFINITIONS

Refer to Chapter One and Chapter Three for a listing of applicable definitions.

4.0 INTERFERENCES

4.1 Method 3052 will overcome the problems associated with incomplete digestion. Incomplete digestion may lead to the incomplete solubilization of sparingly soluble Hg compounds.

4.2 High purity argon (99.999%) must be used as the carrier gas. Nitrogen will reduce the sensitivity by a factor eight-fold, while the use of air will reduce the sensitivity thirty-fold.

4.3 The presence of water vapor in the fluorescence detector may produce scattering effects, positive interferences and degradation of the analytical signal. The use of a dryer tube is required to remove any water vapor from the flow before reaching the detector.

4.4 Contamination is always a potential problem in trace element determinations. See Chapter Three for clean laboratory procedures.

5.0 SAFETY

5.1 Refer to Chapter Three for a discussion on safety related references and issues.

5.2 Many mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Extreme care must be exercised in the handling of concentrated mercury reagents. Concentrated mercury reagents should only be handled by analysts knowledgeable of their risks and of safe handling procedures.

6.0 EQUIPMENT AND SUPPLIES

6.1 Atomic fluorescence system:

6.1.1 Autosampler (optional) - A multi-position computer controlled autosampler may be used. However, it is essential that the autosampler have a wash cycle or "wash pot" to rinse the probe between sampling positions. The autosampler wash water should closely approximate the wash water (Section 7.13) in acid strength.

6.1.2 Peristaltic pump - A three channel peristaltic pump that can deliver reagents and sample at flow rates up to 10 mL/min by varying the pump speed, the pump tubing, or both is required. Silicone pump tubing is required for ppt determinations as PVC pump tubing has been found to adsorb mercury.

6.1.3 Solenoid switching valve - A software controlled valve is required to switch between wash and sample at the proper intervals in the analysis cycle.

6.1.4 Mass flow controllers - Mass flow controllers are required for the carrier and sheath gas flows when analyzing near the detection limit where maximum stability of conditions is critical.

6.1.5 Gas liquid separator - A gas-liquid separator is required to sparge reduced mercury from the liquid stream, direct the mercury vapor and argon carrier gas to the fluorescence detector, and direct the liquid reagents to waste.

6.1.6 Dryer tube - A dryer tube is to be placed in line between the gas-liquid separator and the detector to remove water vapor from the carrier gas stream. Any dryer tube which does not degrade the analysis or sensitivity is acceptable. (PermaPure MD-250-12 or equivalent.)

6.1.7 Fluorescence detector - A fluorescence detector with a high intensity mercury light source and a photomultiplier tube at a right angle to the source is required. Use of 254 nm filter coupled with the chemistry of the stannous chloride reduction in the vapor generator/gas-liquid separator makes the detector highly specific for mercury.

6.1.8 Computer controller - A computer controller and software is required to operate and coordinate the various components of the system and acquire the data as it is produced.

6.1.9 Argon gas supply - High purity argon (99.999%) is required. A gas purifier cartridge is also recommended.

6.1.10 Microwave apparatus. Refer to Method 3051 for a description of an appropriate microwave digestion apparatus.

6.2 Data systems recorder - A recorder is recommended so that there will be a permanent record and that any problems with the analysis can be easily recognized.

6.3 Pipets - Microliter, with disposable tips. Pipet tips should be checked as a possible source of mercury contamination prior to their use. Class A pipets can be used for the measurement of volumes equal to or larger than 1 mL.

6.4 Glassware - All glassware, vessels, pipets, etc., must be very clean. Glass, plastic, and fluorocarbon polymer (PFA or TFM) containers may be used but polymers are not suitable for samples containing metallic mercury. The following is an example of a cleaning procedure successfully used in a trace level laboratory. Soak glassware overnight in a cleaning solution (such as Micro®). Rinse four times with Type I water (ASTM Type I water) and soak overnight in an acid/bromate/bromide mixture. The acid/bromate/bromide mixture is made by adding the bromate/bromide solution from Section 7.9 to dilute acid (approximately 5% v/v) until a yellow color forms (the exact composition is not critical). The container should be covered or closed, as an open container will pick up mercury from the atmosphere and permit bromine vapor to escape to the air. After soaking overnight, add enough 5% hydroxylamine from Section 7.10 to eliminate the yellow color. Rinse six times with Type I water. Cap the glassware tightly if it is not to be used immediately. Store in a reduced mercury atmosphere.

When running samples on a daily basis, vessels require the rigorous cleaning procedure described above every 5 to 7 uses. In between and after each use, the vessels should be soaked in cleaning solution for 2 hours to loosen deposits. They are then cleaned thoroughly with cotton swabs (tested for mercury contamination) and soaked again in cleaning solution overnight. Rinse six times with reagent water. Repeat the soaking and rinsing steps if necessary.

6.5 Balance - A top-loader balance with an accuracy of $\pm$ 0.01 g is required.

6.6 Muffle furnace - A muffle furnace capable of reaching and maintaining a temperature of 150^{o}C is required for purifying the potassium bromate and potassium bromide reagents.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. All reagents should be analyzed to provide proof that all constituents are below the MDLs.

7.2 Reagent water: All references to water in this method refer to reagent water unless otherwise specified. Refer to Chapter One for a definition of reagent water.

7.3 Nitric acid, HNO_3: Use a trace metal grade with negligible mercury content. If the reagent blank is less than the MDL, the acid may be used.

7.4 Hydrochloric acid, HCl: Use a trace metal grade with negligible mercury content. If the reagent blank is less than the MDL, the acid may be used.

7.6 Mercury stock standard solution: A mercury stock solution should be purchased from a reputable source with a concentration of 1.0 mg Hg/mL.

7.7 Potassium bromate (CAS 7758-01-2): Volatilize trace mercury impurities by heating in a muffle furnace at 150°C for at least 8 hours. This procedure is recommended every time the compound is used but if it is stored in a desiccator, ensure that is not contaminated prior to use.

7.8 Potassium bromide (CAS 7758-02-3): Volatilize trace mercury impurities by heating in a muffle furnace at 150°C for at least 8 hours.

7.9 Bromate/Bromide solution: Dissolve 1.39 g potassium bromate and 5.95 g potassium bromide in 500 mL reagent water. Prepare weekly.

7.10 Hydroxylamine hydrochloride (CAS 5470-11-1): Use a source that is specified as suitable for mercury analysis.

7.11 Hydroxylamine hydrochloride solution (5% w/v): Dissolve 2.5 g hydroxylamine in 50 mL of reagent water. Prepare weekly.

7.12 Stannous chloride solution (CAS 10025-69-1), 2% in 10% HCl: Add approximately 500 mL of reagent water to a 1L volumetric flask followed by the addition of 100 mL conc. HCl. Add 20.0 g stannous chloride and stir to dissolve. Dilute to 1 L with reagent water. The solution should be sparged with argon for 30 minutes prior to analysis to remove any traces of mercury. Prepare daily.

7.13 Wash water (reagent blank), 5% HCl: Add approximately 1000 mL of reagent water to a 2 L flask. Add 100 mL conc. HCl and 80 mL of the bromate/bromide solution (Section 7.9). Bring to volume with reagent water. Prepare daily.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See Chapter Three, Inorganic Analytes.

9.0 QUALITY CONTROL

9.1 All quality control data should be maintained and available for easy reference or inspection.

9.2 For each batch of samples processed, method blanks must be carried throughout the entire sample preparation and analytical process according to the frequency described in Chapter One. A method blank is prepared by using a volume or weight of reagent water at the volume or weight specified in the preparation method and then carried through the appropriate steps of the analytical process. These steps may include but are not limited to digestion, dilution, filtering, and analysis. These blanks will be useful in determining if samples are being contaminated. Refer to Chapter One for the proper protocol when analyzing blanks.

9.3 Matrix Spike/Matrix Spike Duplicates (MS/MSDs): MS/MSDs are intralaboratory split samples spiked with identical concentrations of target analytes. The spiking occurs prior to sample preparation and analysis. An MS/MSD is used to document the bias and precision of a method in a given sample matrix. MS/MSDs are to be analyzed at the frequency of one per analytical batch as described in Chapter One. Refer to the definitions of bias and precision, in Chapter One, for the proper data reduction protocols. MS/MSD samples should be spiked at the project-specific action level or when lacking project-specific action levels, between the low and midlevel standards. Acceptance criteria should be set at a laboratory derived limit developed through the use of

historical analyses. In the absence of historical data this limit should be set at ± 20% of the spiked value for precision and ≤ 20 relative percent difference (RPD). After the determination of historical data, 20% must still be the limit of maximum deviation for both percent recovery and relative percent difference to express acceptability.

9.4 For each batch of samples processed, laboratory control samples must be carried throughout the entire sample preparation and analytical process according to the frequency of one per analytical batch as described in Chapter One. The laboratory control samples should be spiked at the project-specific action level or when lacking project-specific action levels, between the low and midlevel standards. Acceptance criteria should be set at a laboratory derived limit developed through the use of historical analyses. In the absence of historical data this limit should be set at ± 20% of the spiked value. Refer to Chapter One for more information.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibration standards - All analyses require that a calibration curve be prepared to cover the appropriate concentration range. Calibration standards are prepared by diluting the stock metal solutions at the time of analysis and digesting them using the same procedure used for actual samples. If running more than one batch of samples during the same week, the microwave-digested standards can be kept in clean dedicated glassware from which dilutions can be made daily which are then prepared with acid and bromate/bromine just as the samples are prepared after microwaving.

10.1.1 Calibration standards must be prepared fresh (or from the weekly microwave digest described in 10.1) each time a batch of samples is analyzed. Prepare a reagent blank and at least three calibration standards in graduated amounts in the appropriate range of the linear part of the curve.

10.1.2 The calibration standards should be prepared using the same type of bromine, acid or combination of acids and at the same concentration as will result in the samples following processing.

10.2 A calibration curve must be prepared each day with a minimum of a reagent blank and three standards. After calibration, the calibration curve must be verified by use of at least a calibration blank and a check standard (made from a reference material or other independent standard material) at or near the mid-range. The calibration curve must also be verified at the end of each analysis batch and/or after every ten samples. The calibration check standard must be measured within 20% of its true value for the curve to be considered valid.

10.3 The working standard curve must be verified by measuring satisfactorily a mid-range standard or check standard and a reagent blank at the end of each analysis batch and/or after every 10 samples. This sample value must be within 10% of the true value, or the previous ten samples must be reanalyzed. The reagent blank must be less than the MDL. If the aforementioned criteria are not met, reanalyze the samples analyzed since the last passing calibration check and calibration blank.

11.0 PROCEDURE

11.1 Prepare samples in a microwave unit using only nitric and hydrochloric acids. Follow instrument manufacturers instructions.

11.1.2 Transfer approximately 1.0 gram of wet sample to a digestion vessel. Add 2.0 mL of concentrated nitric and 6.0 mL of concentrated hydrochloric and cap.

11.1.3 Microwave the samples with a program appropriate for complete digestion. Typically, the temperature should be ramped slowly to 190°C without overpressurization and held at 190°C for 10 minutes or until the digestion is complete.

11.1.4 Cool the samples. During the cooling period, vent and swirl the samples occasionally to release dissolved gases. After the samples have cooled and the dissolved gases have been dissipated, transfer 1.0 mL of the digested sample into a graduated 50 mL centrifuge tube that contains reagent water, 2.0 mL bromate/bromide solution (Sec. 7.9) and 2.5 mL HCl. Bring to volume with reagent water and cold digest for 15 minutes. Document the accuracy of the centrifuge tubes through mass/volume records or use volumetric glassware.

11.2 Set up a software controlled timing sequence for the analysis. Follow your instrument manufacturer's instructions for all settings. Timing sequences that should be addressed are:

11.2.1 Delay: The delay step allows times for the sample line to fill with sample.

11.2.2 Rise: The rise step allows sample to enter the gas/liquid separator and react with the stannous chloride. Mercury in the digested sample is reduced to Hg^0 and the argon carrier stream carries the Hg^0 as mercury vapor to the detector.

11.2.3 Analysis: The analysis time allows for the peak height to rise to its maximum while the software measures the peak height or area.

11.2.4 Memory: The memory time allows the signal to return to the baseline level.

11.2.5 Set the gas flows at a level providing adequate sensitivity for the desired analytical range. Flow rates for the following gases should be established: carrier gas, sheath gas, and dryer tube gas.

11.2.6 When using a variable speed peristaltic pump, choose appropriate sized tubing to obtain an approximate ratio of 2:1 between sample/wash (Sec. 7.13) flow rates and the reductant (Sec. 7.12) flow rate.

11.2.7 Set the gain on the detector to the sensitivity range required for the analysis.

11.2.8 If an autosampler is used, set up a wash solution for the autosampler probes. The autosampler wash solution should closely approximate the wash water (Sec. 7.13) in acid concentration or contain acid at a sufficient strength (typically 5%) to preclude any sample carryover.

11.2.9 Allow 30 minutes for the system to equilibrate before initiating sample analysis.

11.3 Sample Analysis

11.3.1 Add 0.10 mL of the hydroxylamine solution (Sec. 7.11) to remove excess bromine and decolorize the sample.

11.3.2 Allow precipitate or sediment in diluted samples to settle to avoid fouling the valves with solid material during analysis.

11.3.3 Any sample that gives a response greater than the highest standard must be diluted and rerun. Add appropriate amounts of reagents to ensure the reagent concentration of the diluted sample match that of the other samples and the wash (Sec. 7.13).

11.3.4 Any samples that fall outside the laboratory calculated and derived QC range must be re-digested and reanalyzed.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Maintain an instrument log book that contains all information necessary to reproduce the analytical conditions associated with a sample run.

12.2 Sample calculation:

$$\text{Hg in } \mu g/Kg = \frac{\text{result ng Hg}}{\text{L}} \times \frac{\text{0.05 L final vol.}}{\text{0.001 L dig sample}} \times \frac{\text{0.008 L dig soln.}}{\text{g sample (x \%solids)}}$$

13.0 METHOD PERFORMANCE

13.1 Accuracy: Results for accuracy from the US EPA Region IV laboratory are given in Table 1. Data for liquid reference materials and liquid calibration checks used with this procedure are included along with the sediment and tissue materials.

13.2 Precision: Results for precision from the Region IV laboratory are provided in Table 2.

13.3 The laboratory for which accuracy and precision data are presented here also participated in three intercomparison studies.

13.3.1 In the first study, two sediments and two tissues were analyzed for the National Oceanic and Atmospheric Administration in the Seventh Round Intercomparison for Trace Metals in Marine Sediments and Biological Tissues.

13.3.1.1 The accepted value for the sample identified as Sediment T is 0.107 mg/Kg with an acceptable range of 0.087 to 0.127. The value reported from this laboratory was 0.100 with a standard deviation of 0.003 and percent relative standard deviation of 3.0. The number of labs reporting results for this sample was 32.

13.3.1.2 The accepted value for the sample identified as BCSS-1 is 0.163 mg/Kg with an acceptable range of 0.096 to 0.230. The value reported from

this laboratory was 0.199 with a standard deviation of 0.013 and percent relative standard deviation of.3. The number of laboratories reporting results for this sample was 28.

13.3.1.3 The accepted value for the tissue sample identified as Tissue S is 0.0618 mg/Kg with an acceptable range of 0.0409 to 0.0827. The value reported from this laboratory was 0.0574 with a standard deviation of 0.0047 and percent relative standard deviation of 8.3. The number of laboratories reporting results for this sample was 33.

13.3.1.4 The certified value for the tissue sample identified as NIST 1566a is 0.0642 mg/Kg with an acceptable range of 0.0575 to 0.0709. The value reported from this laboratory was 0.0631 with a standard deviation of 0.0042 and percent relative standard deviation of 6.8. The number of laboratories reporting results for this sample was 27.

13.3.2 Two sediments and two tissues were analyzed in the Eighth Round Intercomparision for Trace Metals in Marine Sediments and Biological Tissues for the National Oceanic and Atmospheric Administration.

13.3.2.1 The accepted value for the sediment sample identified as Sediment U is 0.55 mg/Kg with an acceptable range of 0.42 to 0.68. The value reported from this laboratory was 0.63 with a standard deviation of 2.7. The number of laboratories reporting results for this sample was 28.

13.3.2.2 The accepted value for the sediment sample identified as BCSS-1 is 0.180 mg/Kg with an acceptable range of 0.109 to 0.251. The value reported from this laboratory was 0.23 with a standard deviation of 0.01 and percent relative standard deviation of 5.7. The number of laboratories reporting results for this sample was 26.

13.3.2.3 The accepted value for the tissue sample identified as Tissue V is 0.0654 mg/Kg with an acceptable range of 0.0462 to 0.0846. The value reported from this laboratory was 0.058 with a standard deviation of 0.006 and percent relative standard deviation of 8.0. The number of laboratories reporting results for this sample was 32.

13.3.2.4 The certified value for the tissue sample identified as NIST 1566a is 0.0654 mg/Kg with an acceptable range of 0.0587 to 0.0721. The value reported from this laboratory was 0.066 with a standard deviation of 0.003 and percent relative standard deviation of 5.2. The number of laboratories reporting results for this sample was 28.

13.3.3 Ten sediment samples from the Florida Everglades were analyzed and the results from two laboratories are presented in Table 3.

13.4 Comparison data for this method (CVAF) versus cold vapor atomic absorption generated within the Region IV laboratory are presented in Tables 4 and 5. Attention should be drawn to the fact that the lowest results in Table 5 are near the limits of detection for the CVAA method, but well within the CVAF range, while the higher results are obtained by diluting samples for the CVAF method, but are well within the range for the CVAA method. Therefore, the CVAA

method may be more appropriate for the samples with higher levels of mercury and the CVAF method is more appropriate for lower level samples.

13.5 The following documents may provide additional guidance and insight on this method and technique:

13.5.1 W. Van Delft and G. Vos, *Analytica Chimica Acta*, 209 (1988) 147-156.

13.5.2 "Yorkshire Water Methods of Analysis", 5th Edition, 1988. (ISBN 090507236)

13.5.3 "Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

13.5.4 "OSHA Safety and Health Standards, General Industry", (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206, revised January 1976.

13.5.5 "Proposed OSHA Safety and Health Standards, Laboratories", Occupational Safety and Health Administration, FR July 24, 1986.

13.5.6 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.

13.5.7 Standard Methods, 18th Edition, 1992.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society, Department of Government Relations and Science Policy, 1155 16th Street, NW, Washington, D.C. 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Section 14.2.

16.0 REFERENCES

1. “Method for Total Mercury in Drinking Water, Surface, Ground, Industrial and Domestic Waste Waters and Saline Waters”, P.S. Analytical Ltd., Sevenoaks, Kent, U.K.

2. “Method for the Determination of Ultra Trace Level Total Mercury in Sediment and Tissue Samples by Atomic Fluorescence Spectrometry”, EPA Region IV.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain Tables 1 through 5 and a method procedure flow diagram.

TABLE 1

RECOVERY DATA FOR SEDIMENT AND TISSUE REFERENCE MATERIALS, SPIKES, METHODS CHECKS (MC), AND CALIBRATION CHECKS

Matrix	True value[1]	Avg. % Rec.	# of samples	Std dev
NIST 8406 SED	60 µg/Kg	103.3	70	9.3
NRCC BEST1 SED	92.0 µg/Kg	100.9	21	7.4
Sediment spike	22.7 to 68.9 µg/Kg	89.2	38	13.7
NIST 1575 Plant tissue	150 µg/Kg	95.6	13	7.5
NIST 1566 Oyster tissue	84.2 µg/Kg	96.7	72	12.3
NRCC DORM-1 Fish tissue	798 µg/Kg	108	9	8.1
NBS 1641B Inorg water (MC)	38 to 60.8 ng/L	95.6	15	6.4
NBS 1641C Inorg water (MC)	36.8 to 73.5 ng/L	105.6	6	4.3
EPA WS024 Inorg+org water	43.2 to 108 ng/L	88.2	16	7.0
EPA WS029 Inorg+org water	10.1 to 50.6 ng/L	97.3	45	7.3
EPA WS030 Inorg+org water	43.2 ng/L	100.6	24	8.6
EPA WS031 inorg+org water	9.08 to 45.4 ng/L	100.8	49	11.1
Calib checks water	20.0 to 100 ng/L	101.5	139	4.2

[1]True values analyzed at various dilutions.
Source: Reference 2

TABLE 2

PRECISION DATA FOR SEDIMENT AND TISSUE SAMPLES[1]

Replicates	Avg %RSD	# Of samples	Std dev
Sediment original	10.2	280	9.2
Sediment duplicate	8.8	43	8.8
Sediment original vs duplicate	7.5	43	7.0
Tissue original	11.4	61	10.5
Tissue duplicate	12.0	13	10.6
Tissue original vs duplicate	5.7	13	4.7

[1]All samples analyzed twice.
Source: Reference 2.

TABLE 3

EVERGLADES SEDIMENT SAMPLE COMPARISON DATA (µg/Kg)

Sample	Reference Laboratory			Region IV Laboratory	
	Rep 1	Rep 2	Fluorescence Rep 1	Fluorescence Rep 2	Method 245.5
1	11	13	8.6	10.3	
2	6		5.6	5.4	< 25
3	76		76	73	68
4	58		63	64	59
5	410		458	444	424
6	296		501	463	453
7	42		48	46	
8	40		47	58	
9	36		37	37	
10	34	40	42	39	

Source: Reference 2

TABLE 4

COMPARISON OF CVAF METHOD VS. CVAA METHOD 245.5 RESULTS
ON EVERGLADES SEDIMENTS, PEAT AND MARL (µg/Kg)

SAMPLE	AVG	%RSD	CVAA	CVAA %RSD
1	169	2.35	148	11.8
2	197	0.35	248	20.3
3	89	8.04	46	56.6
4	64	6.21	48	26.0
5	28	3.09	23	16.4
6	56	3.76	47	15.4
7	97	2.88	75	22.6
8	172	0.11	161	5.7
9	79	7.89	71	10.0
10	75	4.52	68	8.3
11	79	0.70	76	2.8
12	99	1.54	81	18.1
13	67	1.97	58	12.8
14	146	19.25	130	10.6
15	63	0.52	59	6.3
16	157	0.10	140	10.7
17	144	2.37	144	0.2
18	450	2.55	424	5.5
19	482	6.99	453	5.6
20	247	2.89	226	7.7
21	161	1.97	195	17.8
22	108	4.76	118	7.6
23	278	15.96	356	22.0
24	278	13.71	175	40.0
25	273	2.68	209	23.5
26	456	7.79	285	41.1
27	136	6.83	128	5.42
28	263	3.95	215	17.9
29	89	0.75	80	9.3
30	108	4.79	119	8.4

Source: Reference 2

TABLE 5

COMPARISON OF CVAF METHOD VS. CVAA METHOD 245.5 RESULTS
ON TISSUE SAMPLES (µg/Kg)

Sample	Avg	% RSD	CVAA	%RSD CVAA
Alligator Liver 1	802	18.7	950	15.0
Alligator Liver 2	133	19.9	170	21.1
Alligator Liver 3	216	5.2	230	5.5
Alligator Liver 4	62.7	4.6	72	11.7
Gambusia 1	38.2	6.4	66	47.4
Gambusia 2	165	26.7	120	28.2
Mixed Fish Comp.	70.1	10.6	74	3.4
Bass Filet 1	460	22.2	370	19.4
Bass Filet 2	987	5.5	910	7.2
Bass Filet 3	559	15.9	560	0.2
Bass Filet 4	274	52.8	202	26.9
Bass Filet 5	172	2.6	155	9.5
Catfish Filet 1	62.2	12.3	80	22.3
Catfish Filet 2	133	0.5	150	10.4
Catfish Filet 3	119	7.1	130	7.9
Bluegill Filet 1	36.4	5.1	37	1.6
Bluegill Filet 2	53.7	0.0	36	35.1
Clam Tissue 1	28.5	87.1	25	11.8
Clam Tissue 2	21.1	11.6	30	31.0
Clam Tissue 3	17.6	12.2	30	46.3

Source: Reference 2

MERCURY IN SEDIMENT AND TISSUE SAMPLES BY ATOMIC FLUORESCENCE SPECTROMETRY